

Optical Sensor Technology Development and Deployment

Federal Manufacturing & Technologies

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Department 833

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Final Report on Enhanced Surveillance 708038
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Abstract

The objectives of this ESP (Enhanced Surveillance) project are to evaluate sensor performance for future aging studies of materials, components and weapon systems. The goal of this project is to provide analysis capability to experimentally identify and characterize the aging mechanisms and kinetics of Core Stack Assembly (CSA) materials. The work on fiber optic light sources, hermetic sealing of fiber optics, fiber optic hydrogen sensors, and detection systems will be discussed.

Summary

Room temperature changes were found to be the cause of most drift and noise problems with the hydrogen sensor system. The use of fiber optic spectrometers was abandoned due to these problems and a Fiber Optic Hydrogen Sensor (FOHS) system was found to be more stable for this application. The FOHS system was redesigned and has eliminated the effects of room temperature changes on its response. SMA 905 connectors, also a source of noise, were eliminated except inside of the temperature-controlled FOHS system. The lens holder was redesigned for use with off-the-shelf standard VCR components and eliminates the need for manual lens focusing. A modeling simulation was used to verify that the new design was insensitive to thermal changes. The lens coatings were more thoroughly characterized and several tests were established for monitoring production variability. A commercial source of coated lenses was established and response output was more than doubled.

Discussion

Scope and Purpose

The objective of KC10 is to evaluate sensor performance for future aging studies of materials, components and weapon systems. In addition hydrogen, temperature, pressure and moisture sensors for measuring and recording data in real time will be developed and their performance characterized. Aging canister sensing systems will be evaluated and their system performance in aging tests will be demonstrated.

A flow diagram showing the separate portions of the hydrogen sensor system is shown in Figure 1.

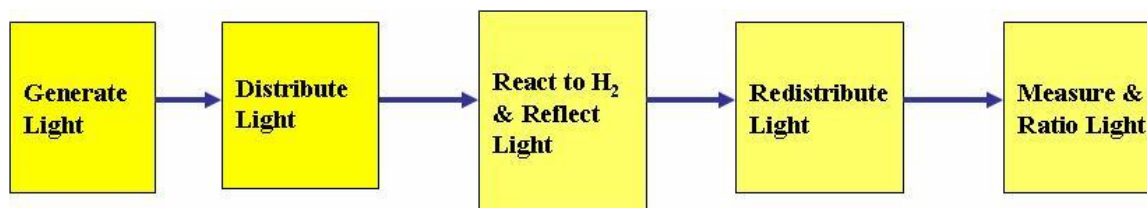


Figure 1. Flow Diagram of Hydrogen Sensor System

Two separate data collection systems are used. One computer collects the pressure and temperature data as a function of time. The second computer controls the operation of the spectrometer, multiplexer and data collection, (percent transmission), as a function of time. The rest of this report will discuss in detail the various portions of this data measurement and collection system.

Activity

Light Generation

Tungsten Halogen Light Sources

Several different light sources have been evaluated for use in hydrogen detection. The initial tungsten halogen light source was contained inside the Savannah River Technical Center (SRTC) spectrometer. The second SRTC spectrometer had an external light source with an electronically controlled shutter and is shown in Figure 2. A third calibrated tungsten halogen light source shown in Figure 3 was obtained from Ocean Optics.



Figure 2. SRTC Tungsten Halogen Light Source



Figure 3. Ocean Optics Tungsten Halogen Light Source



Figure 4. StellarNet Tungsten Halogen Light Source

A typical spectral output from a tungsten halogen light is shown in Figure 5.

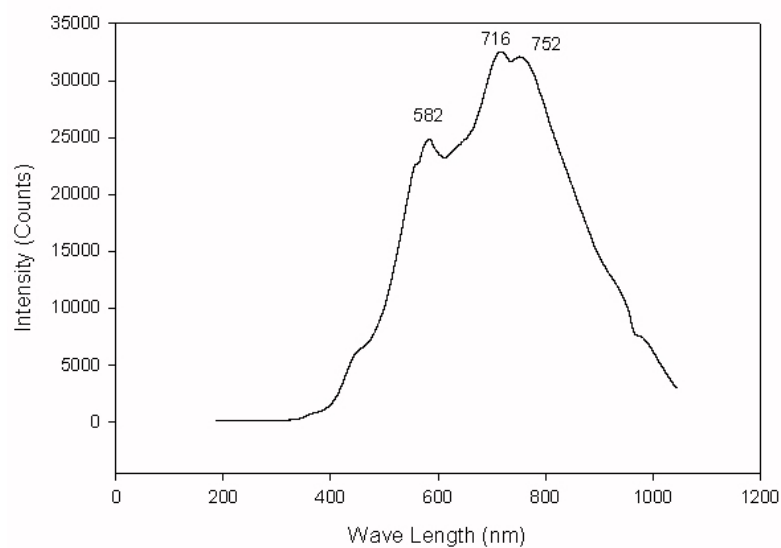


Figure 5. Typical Light Output from a Tungsten Halogen Light Source

Deuterium/Halogen

A deuterium/halogen light source is shown in Figure 6.



Figure 6. Deuterium/Halogen Light Source

This Duolite light source by Equitech has a halogen source that is focused through a switchable deuterium source.

Light Emitting Diodes

The next type of light source evaluated was light emitting diodes (LEDs). An example of an LED light source supplied by INFOS is shown in Figure 7.

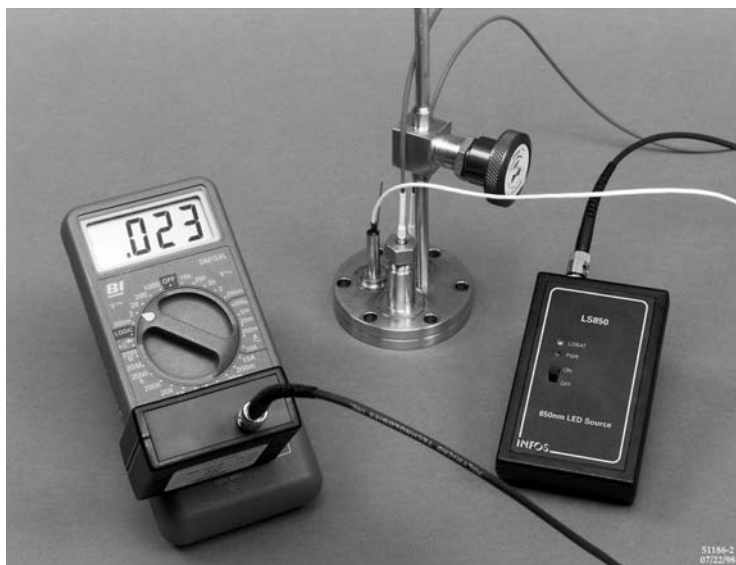


Figure 7. LS850nm LED Light Source

The light source is shown on the right, a photo detector on the left and a retort head assembly in the center. This INFOS source detector system is used to adjust the lens position for maximum light return.

All of the light sources suffer a problem of light intensity stability to some degree or another. The light source output intensity is proportional to the current input and the temperature of the light source. Figure 8 shows the effect of temperature on a tungsten halogen light source.

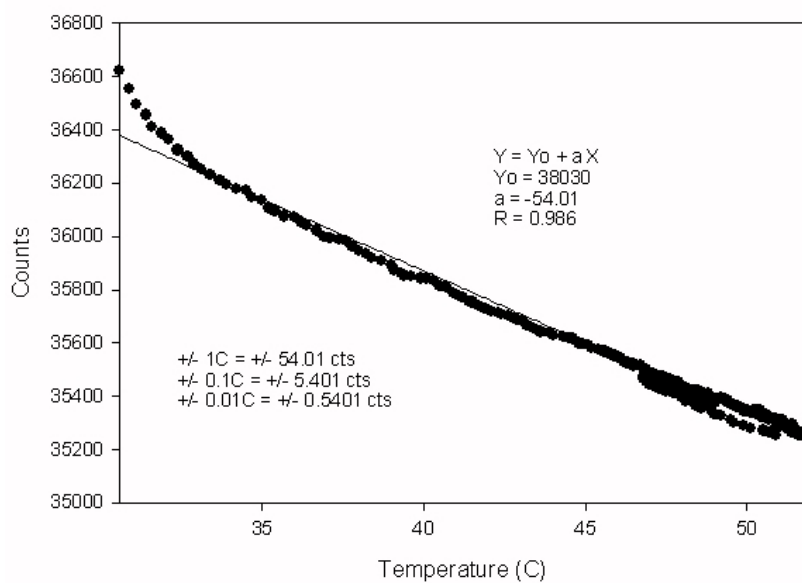


Figure 8. Light Output vs Temperature for a Tungsten Halogen Light Source

The effect of voltage and temperature on an LED output is shown in Figures 9 and 10.

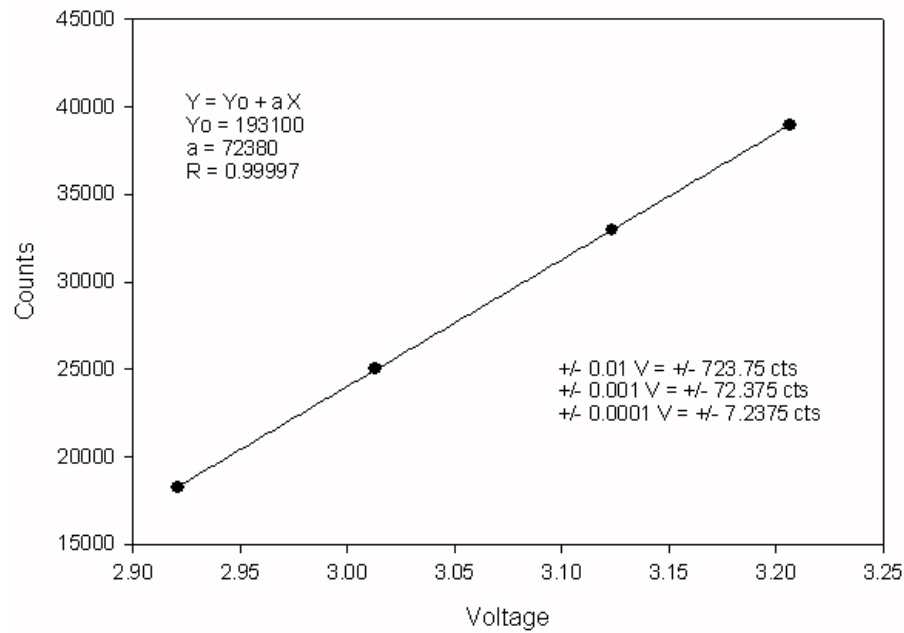


Figure 9. LED Intensity vs Voltage

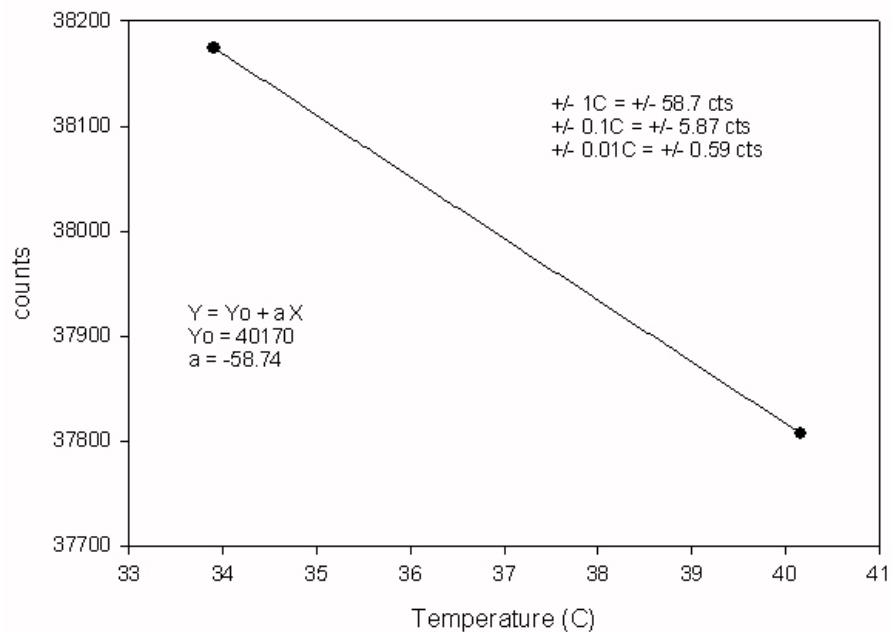


Figure 10. LED Intensity vs Temperature

The LED chosen for use in the final FOHS system that will be discussed later is shown in Figure 11.



Figure 11. Opto Diode Corporation OD 880 FHT LED

LED: OPTO DIODE CORP OD880FHT

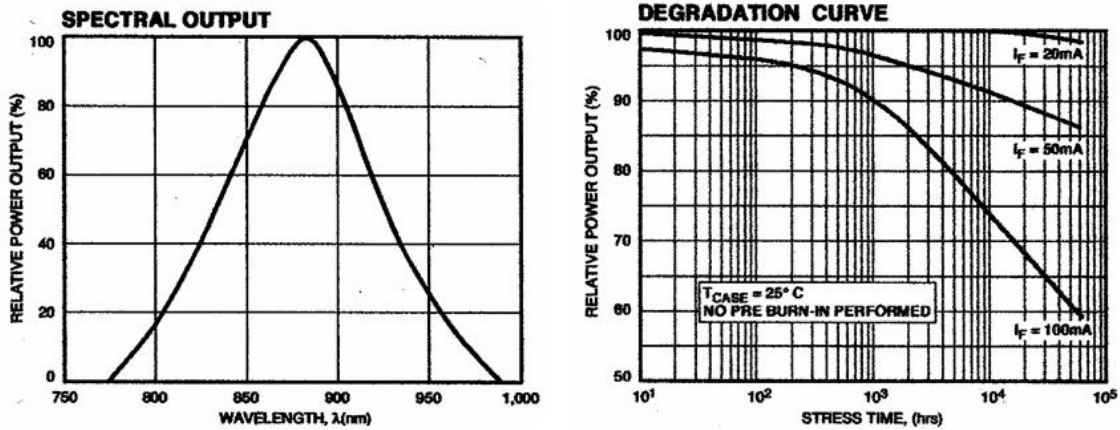


Figure 12. Spectral Output and Degradation Curves for OD 880 FHT LED

Xenon Strobe Flash Light

The original EPP3000 spectrometer from StellarNet will be discussed later. This spectrometer was supplied with the Xenon strobe flash light source shown in Figure 13. This light source was not stable for long-term tests. Drift in the light intensity was not acceptable. This light source was replaced with a blue LED light source which was much more stable.



Figure 13. Xenon Strobe Flash Light Source

Laser Diode

The need was identified for a light source that would provide constant output intensity over time and temperature for use with the hydrogen sensor tester and the new portable hydrogen sensor system. A fiber coupled laser diode with temperature compensation capabilities was identified as an alternate light source for evaluation. This light source proved to not be stable enough for this application. Details of the performance can be found in Topical Report KCP-613-6365¹.

Temperature Compensated Light Source

A temperature compensated light source was desired as input to the spectrometer. Prior experiments had utilized an uncompensated white light source employing a tungsten filament lamp. A solid-state light source alternative was attractive because of its smaller size, longer working life, and ease of temperature compensation. Details of the temperature compensated light source can be found in Topical Report KCP-613-6365¹. The use of temperature compensated LED light sources was carried over into the FOHS system that will be discussed later.

Light Distribution

Once a stable light source is achieved, the light must be directed to the hydrogen sensitive coating and then reflected back to a detection system. The light was transported by 400 micron fiber optic fibers. SMA 905 bulkhead connectors, shown in Figure 14, were used to connect two fibers together.



Figure 14. SMA 905 Fiber Optic Connectors

The standard SMA 905 connector is made from chrome plated brass. These connectors were found to cause drift in light intensity due to room temperature fluctuations. SMA 905 type connectors were machined from Kovar and Invar along with β -eucryptite filled epoxy to try to eliminate this problem. The changes did reduce the effect but did not eliminate it. The final approach was to thermally stabilize the connectors. The thermal electrically controlled bulk head connectors are shown in Figure 15.

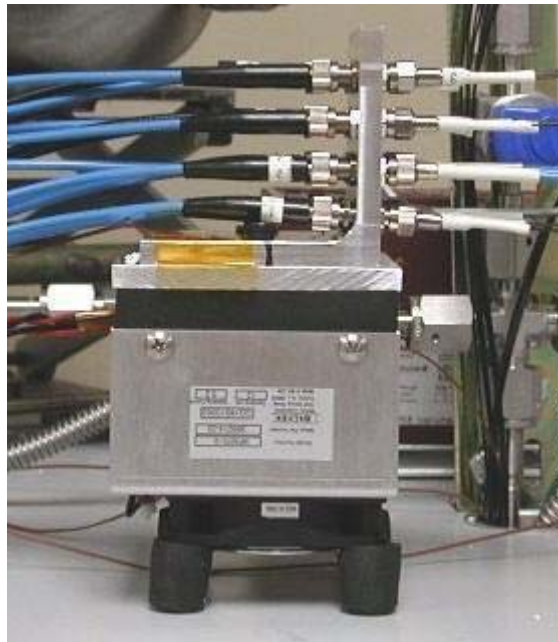


Figure 15 Thermally Controlled SMA 905 Connectors

Since the original systems were single channel fiber optic spectrometers built by SRTC, and multi channels were desired, the use of fiber optic multiplexers was evaluated.

Terahertz Multiplexer

A Terahertz model FM-1600 Optical Switcher with 400 μ m fibers was used to multiplex the light source and spectrometer between different CSAs. The multiplexer is shown next to the SRTC spectrometer in Figure 16.

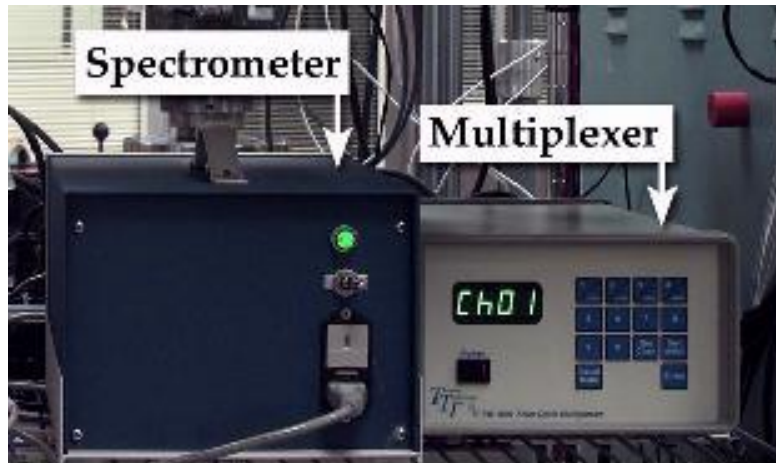


Figure 16. 2 X 8 Fiber Optic Multiplexer.

A laptop computer via the RS232 serial port controlled operation of the multiplexer. Several problems were encountered with the multiplexer. At times physical alignment of fibers while switching from one channel to another would be off, causing step changes or noise in the baseline. In addition, long-term running of the multiplexer would result in a lock-up condition, requiring a restart of the computer.

FM&T Multiplexer

Due to problems with lock-up and alignment, an alternate multiplexer design was investigated. The design of the multiplexer can be understood with reference to Figure 17. Details on the construction and testing of this multiplexer are discussed in Topical Report KCP-613-6365¹.

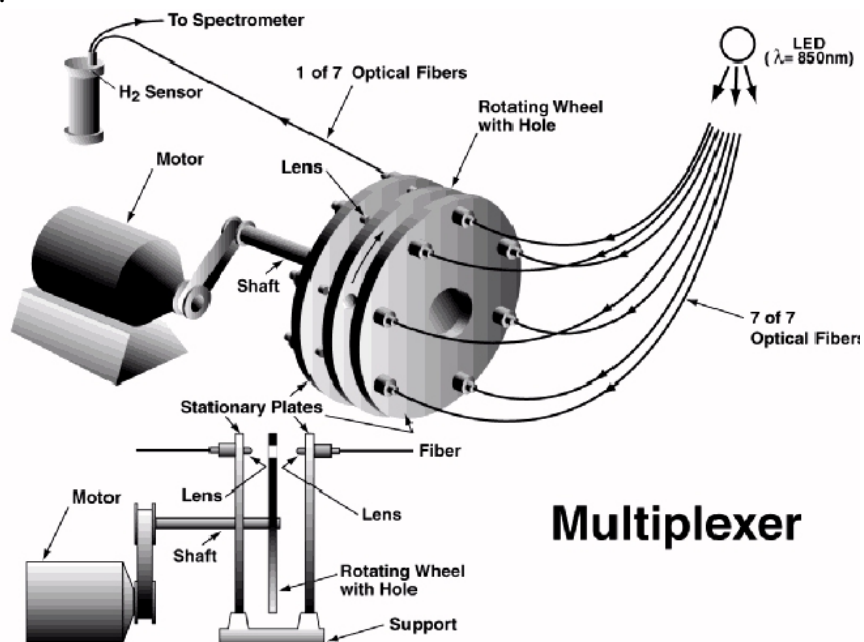


Figure 17. FM&T Multiplexer

Light Octopus

Options to not using a multiplexer but still monitoring multiple sensors are to use multiple light sources and detectors or a single light source that is split into multiple fibers and multiple detectors. The term referred to for the multiple fiber light cable is a “fiber optic octopus.” Examples of these multi-channel light sources are shown in Figures 18 and 19.

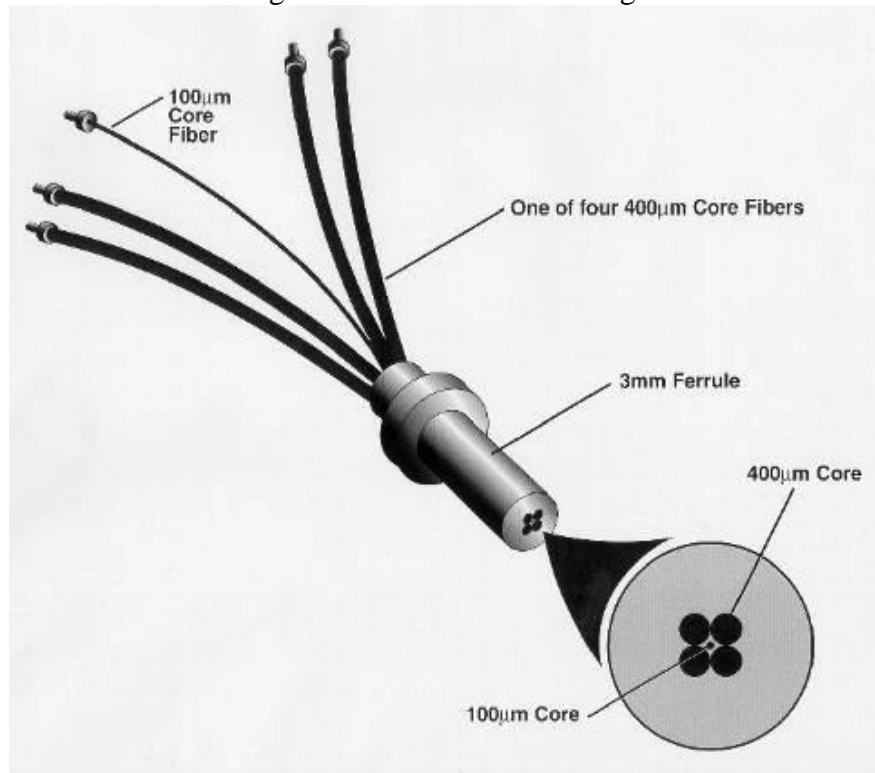


Figure 18. 4-Channel Light Octopus with an Additional Reference Fiber



Figure 19. 8-Channel Fiber Optic Octopus

Once the light reaches the retort where the hydrogen is to be measured, it must enter into the retort, be reflected off the sensor and exit back out of the retort. This must be accomplished while maintaining retort hermeticity.

Hydrogen Sensors

Retort Chamber/Fiber Cable Assembly

The original retort chamber assembly is shown schematically in Figure 20. The portion of the assembly used to hold the special sample under test is cylindrical with an inner diameter of 1.375 inches and a length of 5.0 inches. The bottom of the chamber is sealed hermetically by a steel plate with a conflat knife-edge seal. The upper end of the cylindrical sample chamber is sealed with a specially machined flange plate having a conflat knife-edge seal identical to that at the bottom. The machined flange plate accommodates an access tube, a pressure sensor, and two ports for fiber optic cable connections to the chamber. The stainless steel access tube is laser welded in place, and it provides access to an external vacuum system, but can also be used to introduce argon and nitrogen purging gases to the chamber. The steel tube is fitted with a Nupro valve for closing off the chamber from the external system

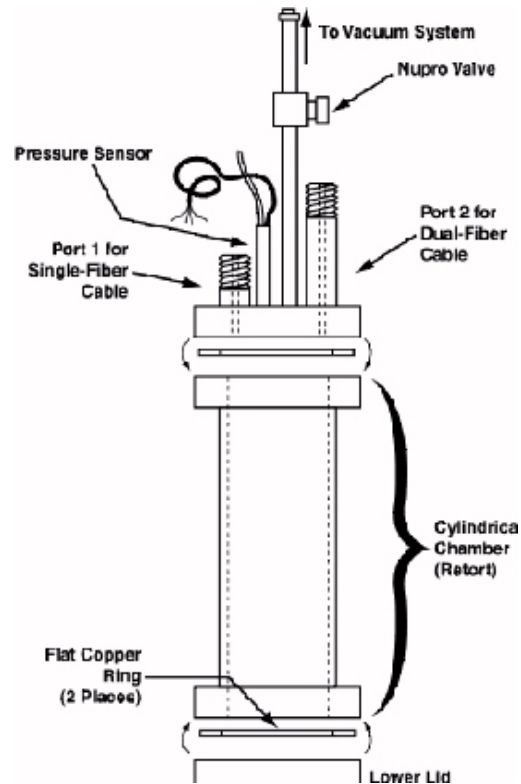


Figure 20.. Retort Chamber Assembly

The fiber optic cables require hermetic seals to the flange plate. This was accomplished by sealing the optical fiber in the end of a Kovar connector, which in turn was sealed to the flange

port with a metal O-ring. The technique for making the hermetic seal of the fiber in the end of the Kovar connector can be understood with reference to Figure 21. The end of the machined Kovar connector contains a small hole that matches closely the diameter of the silica fiber. Concentric with the hole is a counter bore, which receives a small tube of sealing glass, slipped over the fiber. A lens is used to focus a CO₂ laser beam on the end of the Kovar ferrule, and the connector/fiber-cable combination is rotated about its axis to achieve uniform heating of the end.

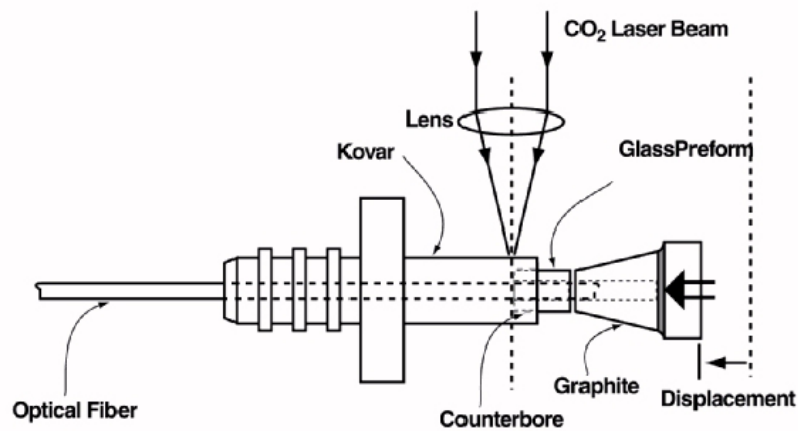


Figure 21. Hermetic Seal of Fiber in Kovar Connector

Some details of this are given in the schematic view of Figure 22. In this case, the glass preform was chosen to have an inner diameter just large enough to encircle the two fibers.

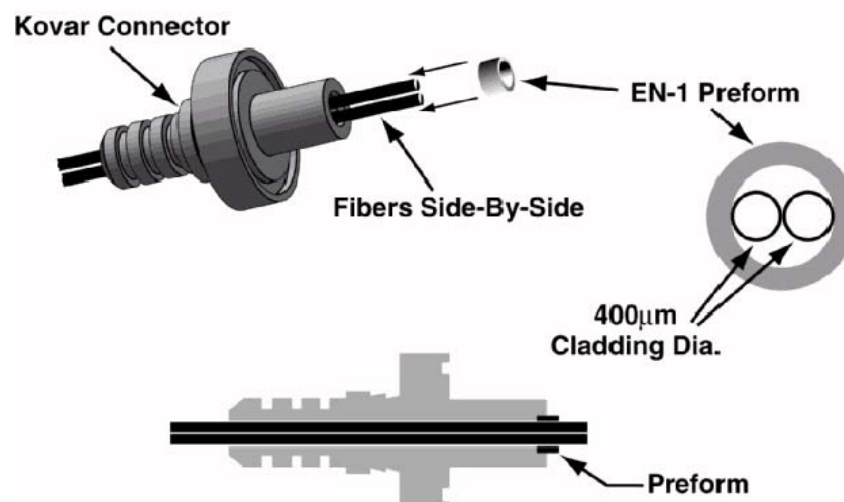


Figure 22. Configuration for Hermetic Seal of Two Fibers Side-By-Side in Kovar Connector

Prior to assembly of the fiber optic cables on Port 1 and Port 2 of the flange, each cable end was tested individually for hermeticity. Details of the leak testing can be found in Topical Report KCP-613-6365¹.

Second Generation Retort Chamber Assemblies

Two changes were made to the original design. First, both ports were designed for dual-fiber cables. Secondly, the hermetic seal of the fiber optic connector to the flange was changed from a metal O-ring to a Varian conflat knife-edge. This was in effect a scaled-down version of the large conflat seals at top and bottom of the cylindrical retort chamber. Details of the knife-edge seal on the Kovar connector for mating with the knife-edge seal on the port on the flange are shown in the sketch of Figure 23.

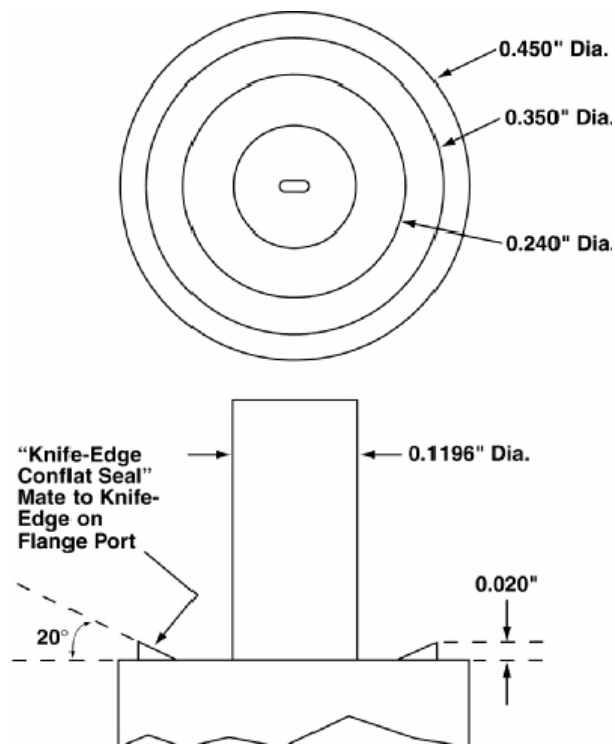


Figure 23. Knife Edge Seal

Details of the leak testing of this design can be found in Topical Report KCP-613-6365¹.

Hydrogen Sensor Lens Holder Redesign

The lens holder was redesigned to include several improvements. The holder was designed to use as many commercially available parts as possible. A standard VCR type vacuum seal was used with off-the-shelf VCR fittings and copper sealing washers. Manual focusing was eliminated. Prior to manufacture, a thermal simulation model was used to finalize the new sensor assembly. The model was successful in ensuring there would be no change in the focus of the sensor lens as a result of temperature fluctuation. This new design is shown in Figure 24. This component is critical in meeting performance specifications in support of Y-12 and LLNL development ESC activities.

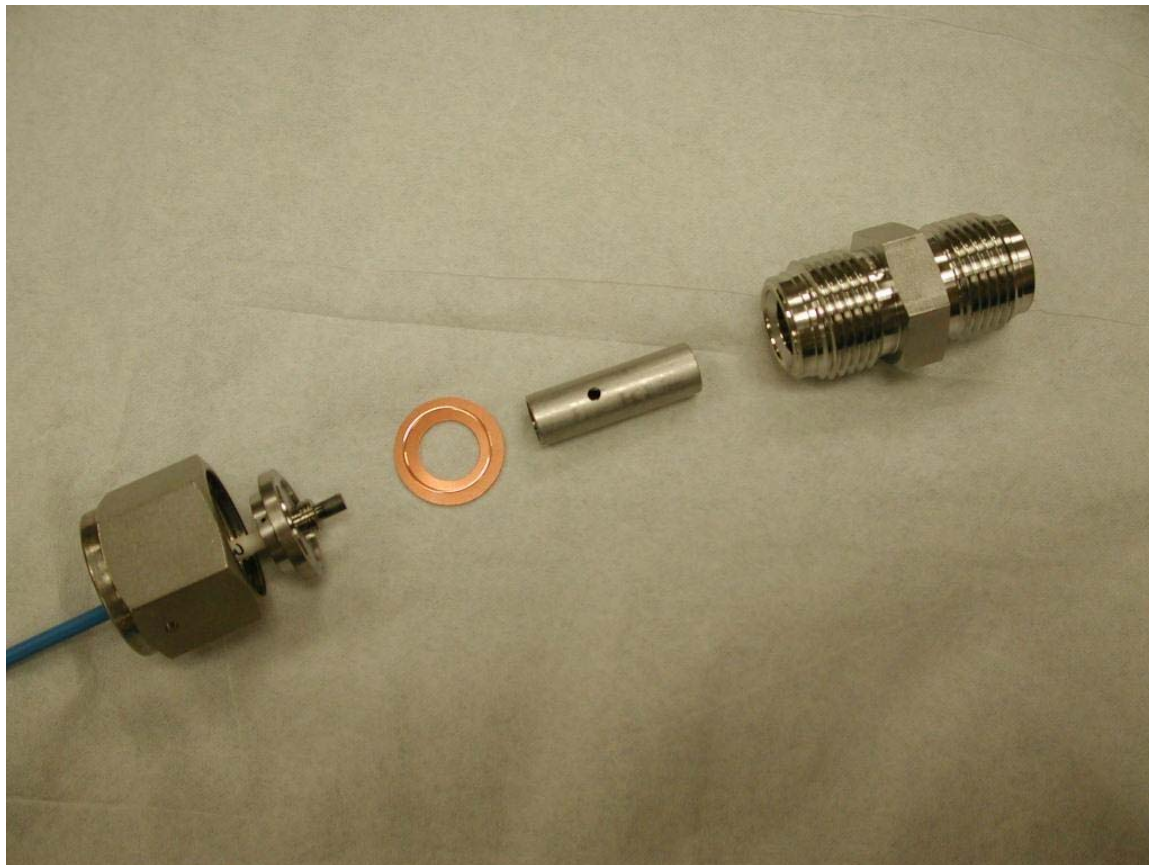


Figure 24. VCR Type Lens Holder Assembly

Hydrogen Sensor Lenses

In order to prevent delamination of the sputtered metallic coatings from the lens materials shown in Figure 25, a thin layer of nickel or chromium is first applied to the lenses. This layer is typically 50 angstroms or less. Next, the active layer that responds to hydrogen is applied. Finally, a protective layer is sometimes applied.

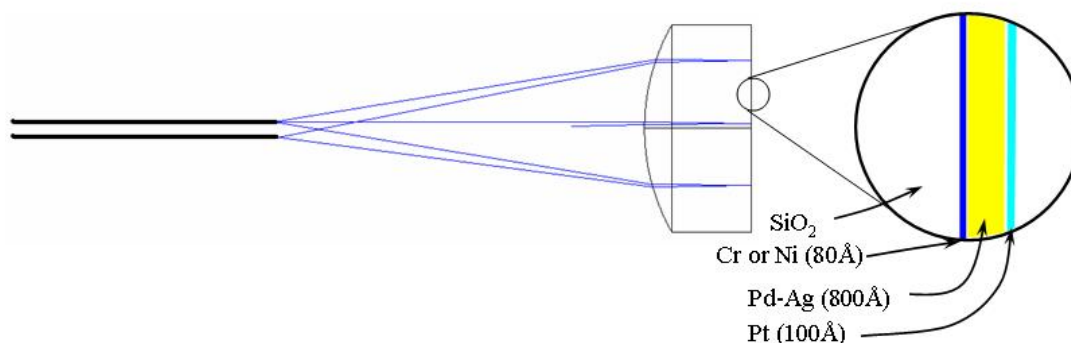


Figure 25. Lens Coatings

Coating compositions for SRTC, Sandia and NexGen are discussed in Topical Report KCP-613-6365¹.

A Designed Experiment (DOE) was used to investigate the effects of sensor coating thicknesses on sensor performance and response. As shown in Figure 26, the sensor response to hydrogen is insensitive to coating thickness for 750 to 1250 angstroms Pd/Ag.

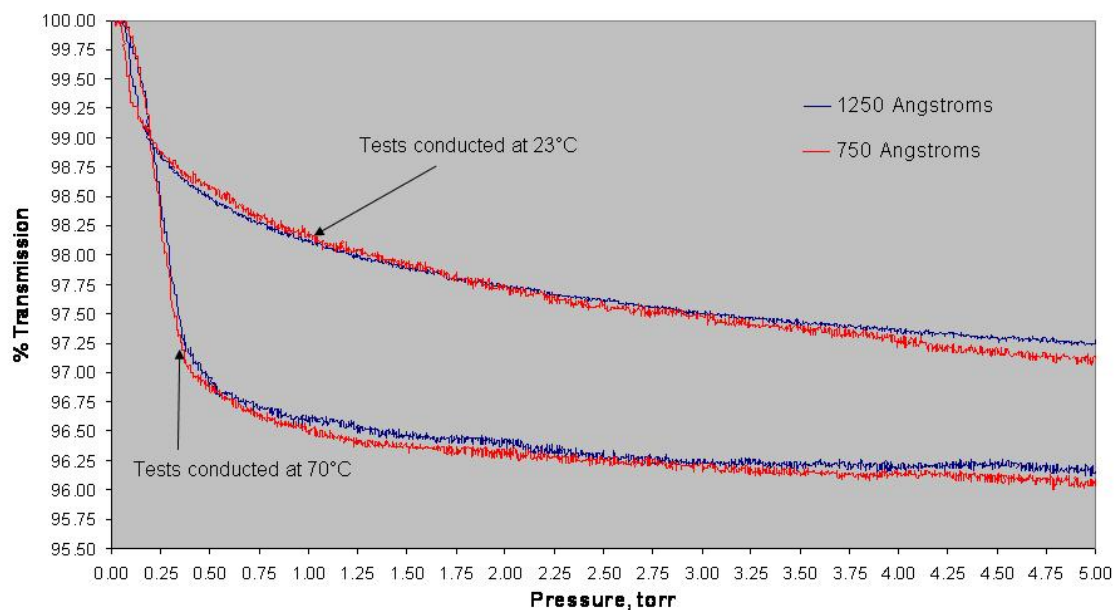


Figure 26. Effects of Sensor Coating Thicknesses on Performance and Response

Vendor sensors showed a larger response to hydrogen than the sensors coated at KCP tested so far (92% transmission vs. ~96% transmission, respectively). This difference can be seen in Figure 27. The KCP sensors were fabricated using a 60/40 palladium/silver target while the vendor supplied sensors were fabricated with a 70/30 target.

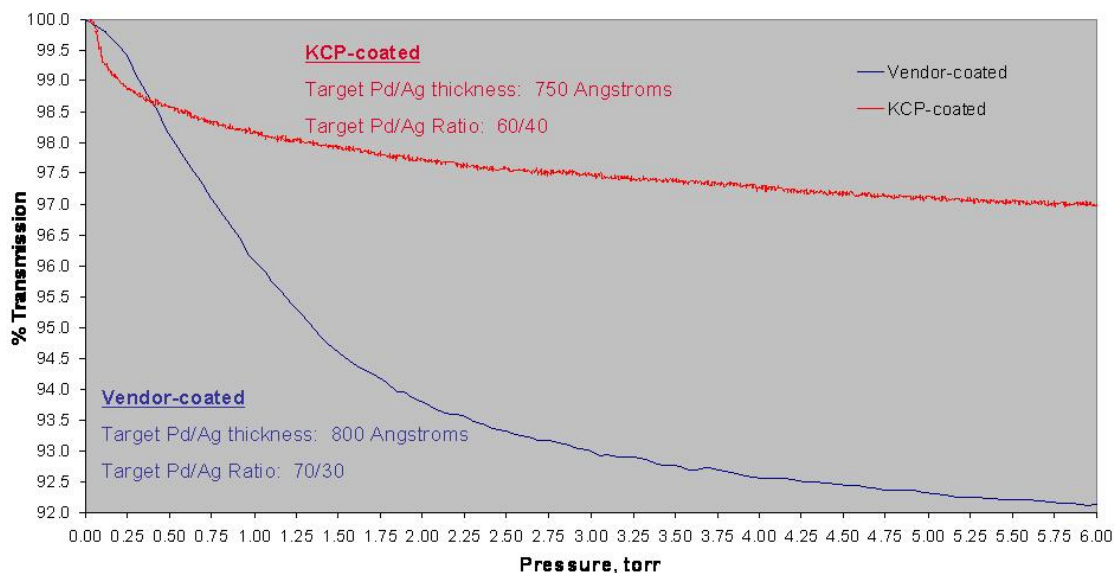


Figure 27. Differences in Response of Vendor vs KCP Coated Lenses

A set of additional tests was established as a method to ensure sensor repeatability. The following tests will be run on all new sensors when they are manufactured:

SEM analysis

Visually inspected under an optical microscope

Surface roughness

Auger analysis

Adhesion pull test

Reflectivity, transmission and absorption, scattering at 1.06 microns.

Fifty BK7 witness samples (1" diameter by 0.125" thick) were obtained for use in KCP's coating chamber as coating verification samples. The thicker substrates do not deform with our applied coating and allow more accurate stylus profilometer measurements of the coating thickness to be performed.

A surface RI quick test was performed to evaluate the scratch resistance/hardness of the H₂ sensor coatings. Samples were photographed under a microscope before and after cleaning with spectroscopic grade alcohol and a high quality lint-free lens tissue. The lens tissue utilized meets Fed. Spec. NNN-P-40B Type 1, Class 1, for silicon free, lint free lens tissue. A test was completed, verifying the scratch resistance/hardness of the H₂ sensor coatings. It demonstrated that an alcohol and lens tissue wipe was effective at removing dust particulates and other debris from the coated surface without adding a noticeable number of new scratches.

Surface RMS roughness measurements samples were collected utilizing the Zygo 633nm interferometer in the EB6 Laser-Optics Lab. This data is used to correlate these noncontact measurements with traditional stylus profilometer measurements to be performed by KCP on same witness samples.

Spectrometers

The original spectrometer, light source and lenses used in this project were fabricated by SRTC. Figure 28 shows this first fiber optic spectrometer used at KCP.

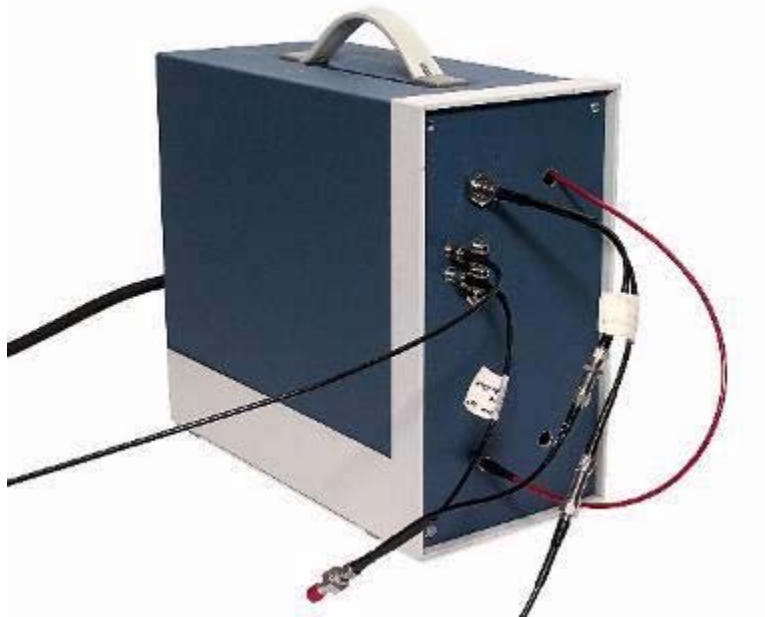


Figure 28. SRTC Fiber Optic Spectrometer

The spectrometer has an internal tungsten halogen filament light source, a Carl Zeiss Model MCS500 spectrometer, dual optical switch and a 488 interface for PC control and data collection. The MCS500's frequency range is 356 to 766 nm with a noise to signal ratio of 0.00005 A.U. RMS. The MCS500 uses a 512-element diode array detector.

The second generation SRTC extended range spectrometer is shown in Figure 29.



Figure 29. Extended Range SRTC Spectrometer

This spectrometer uses a Carl Zeiss model UV-NIR and a National Instruments 16 bit PCMCIA interface card. The spectrometer uses a 1024-element diode array detector with a spectral range from 215 to 1015 nm. The light source and 2 by 8-channel multiplexer in this system are located external to the spectrometer.

Multi-Channel Spectrometers

In 1997 SRTC identified the advantages of an array spectrometer. The StellarNet EPP3000 spectrometer became available in March 1999. The StellarNet EPP3000 array spectrometer also contains an internal 1 to 8-channel xenon light source. The panel rack mount spectrometer is shown in Figure 30.



Figure 30. StellarNet EPP3000 Spectrometers

The figure shows 8-channel versions with 5 and 8-light outputs and 8-channel array inputs. The EPP3000 utilizes a Hamamatsu 64 X 1024 element temperature controlled CCD detector. The spectral range is 190 to 790 nm with a signal-to-noise ratio of 0.00005 A.U. RMS. Integration time can be adjusted from 10 ms to 60 seconds. Communications to a PC is made through an enhanced parallel port with 16 bit A/D electronics.

Several problems were encountered with these fiber optic spectrometers. The original xenon strobe flash light source was not stable and would drift with time. This light source was replaced with a blue LED light source. This light source was much more stable and eliminated some of the drift problems. Again room temperature changes were a problem. Temperature changes affected both the light output and the detector response. The fiber optic spectrometer had options for thermal electric cooling. This was installed and the entire unit was placed in a insulated thermal electrically controlled box, shown in Figure 31. In addition, all power supplies that were sources of heat were located outside of the box.

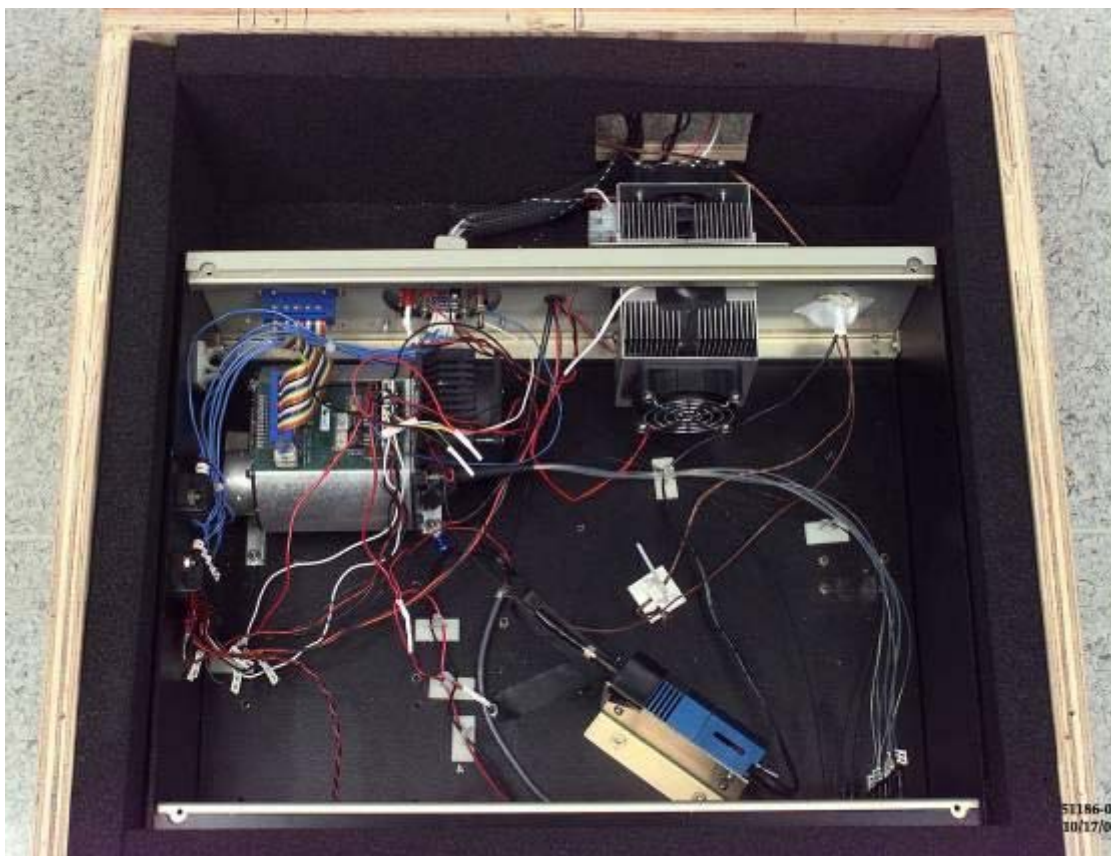


Figure 31. Thermal Electrically Controlled Spectrometer and Light Source

Although these modifications solved the drift problems, an additional problem was encountered. This spectrometer uses a 64 by 1024 array detector. The 64 vertical pixels were divided into 8 by 1024 for each channel. This arrangement resulted in cross-talk between adjacent channels. Several software fixes were tried but were unsuccessful. The next approach was to turn off rows of pixels between the eight channels. This lowered the effect but did not eliminate it. More and more of the adjacent rows were turned off until we were down to a 4-channel system. Since so many of the pixels were being turned off, the signal-to-noise level increased to an unacceptable level.

The next fiber optic spectrometer was the StellarNet SS Rack 8-Channel Spectrometer shown in Figure 32. This system has 8 individual spectrometers on ISA computer cards. Software was developed in house to operate the spectrometer using National Instruments LabView.

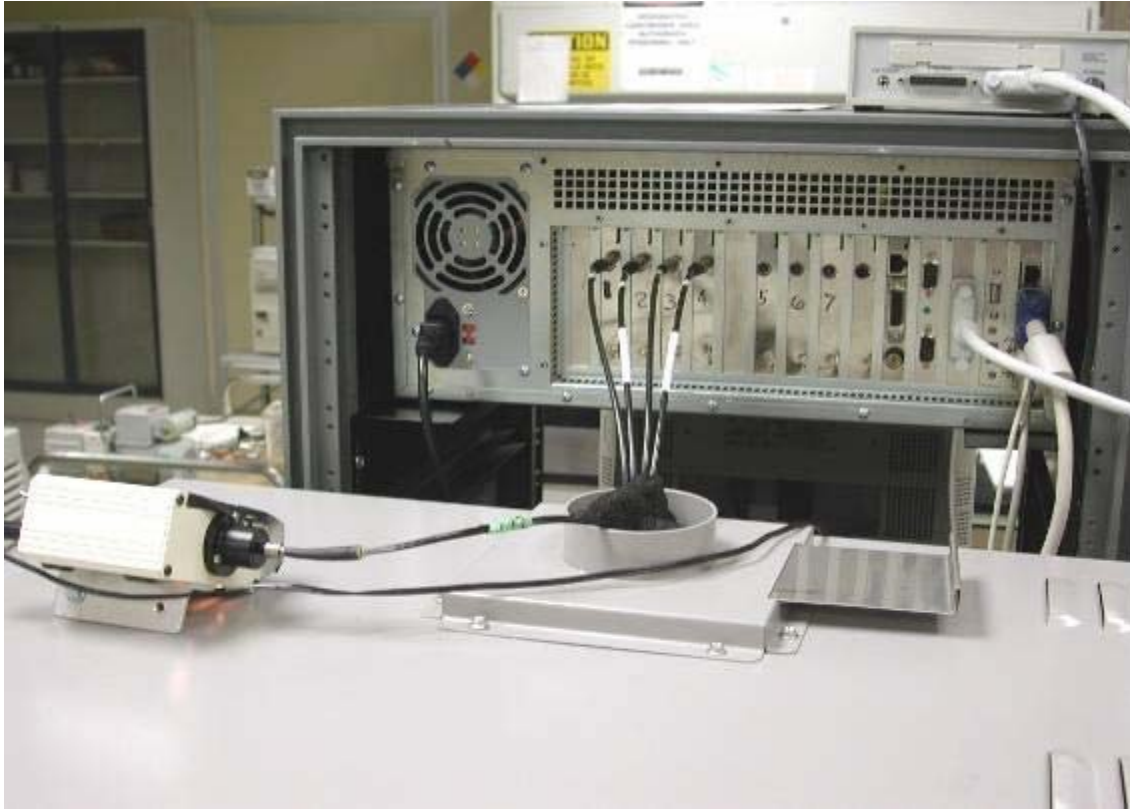


Figure 32. StellarNet SS Rack 8-Channel Spectrometer

Data analysis showed that the IR system experienced drift and noise because it is not thermally controlled and because the reference channel drifted, as shown in Figure 33. This made it more difficult to interpret the results and develop accurate calibration equations for the IR system, so focus was shifted to analyzing the Fiber Optic Hydrogen Sensor System, (FOHS) System, results. A schematic of this system is shown in Figure 34.

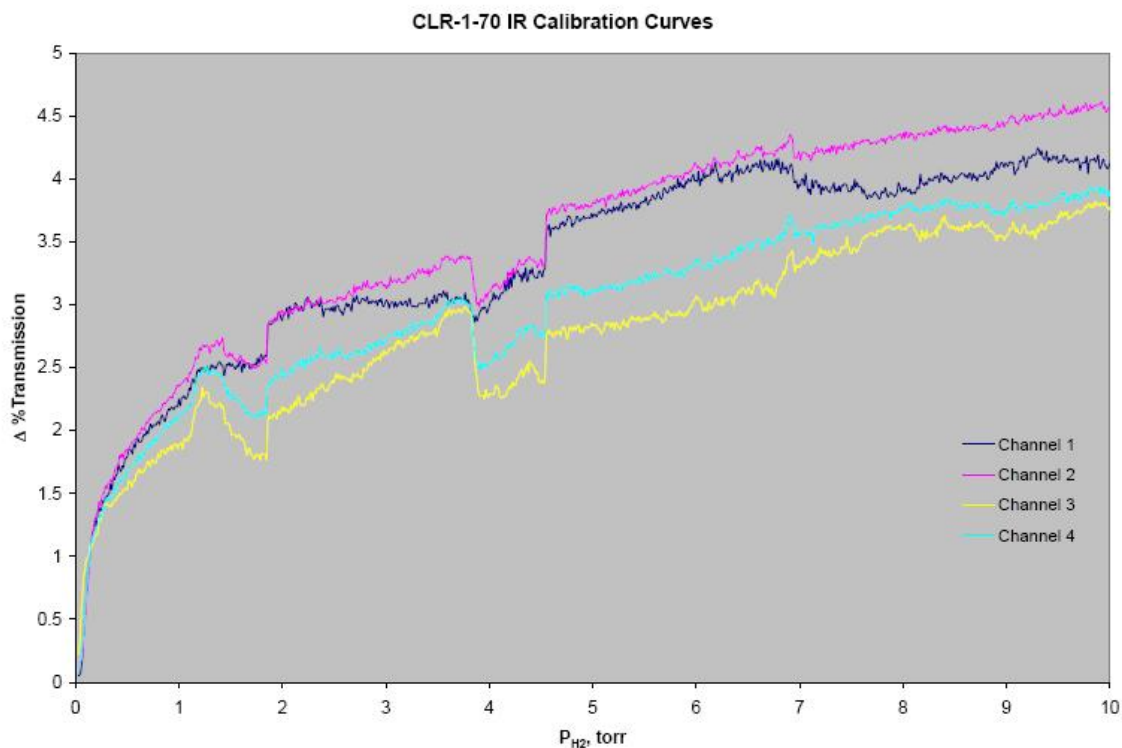


Figure 33. IR Calibration Data

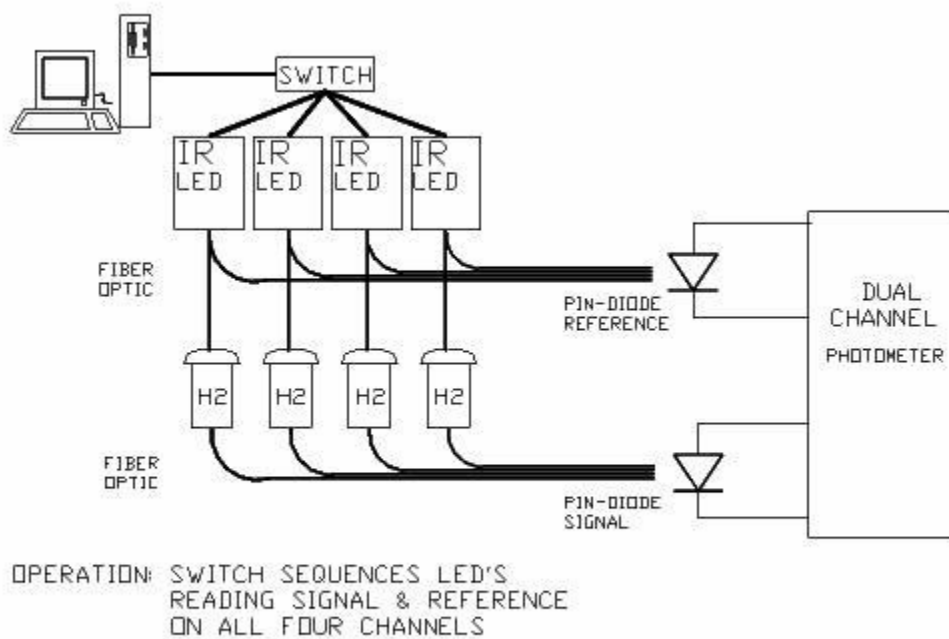


Figure 34. Fiber Optic Hydrogen Sensor System Schematic

During operation of the FOHS system, all LEDs are turned on by a computer program. The LEDs are allowed to stabilize to constant output intensity. During interrogation of a sensor, all of the other LEDs are turned off. After the reading is taken, they are turned back on and again allowed to stabilize. This process is repeated for each of the other sensors. The initial FOHS system is shown in Figures 35 and 36.



Figure 35. Top of the Initial FOHS System

In this initial design, commercial blue LED light sources were used and separate thermal electric coolers were used for the LEDs and the photodiode detectors. In Figure 36, the details of the power supplies and thermal electric coolers can be seen. These components were located outside the insulated box.

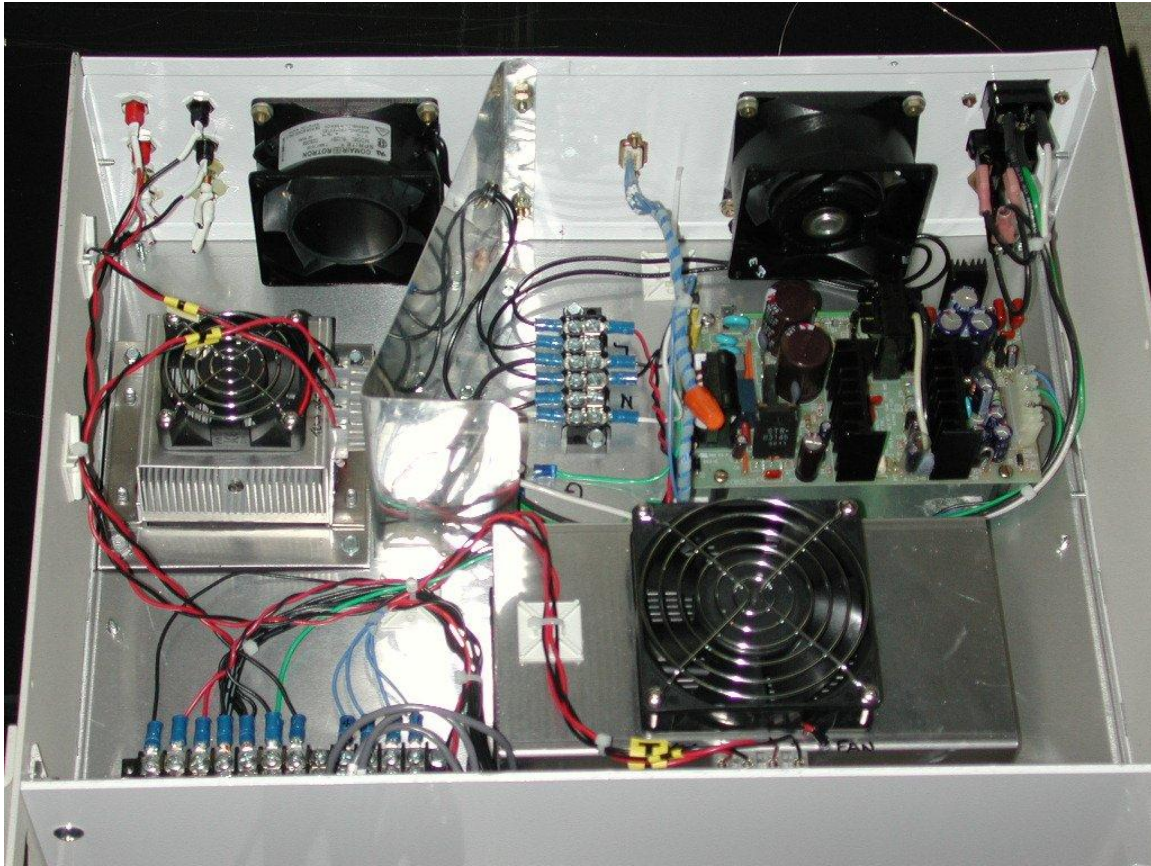


Figure 36. Bottom of the Initial FOHS System

The second FOHS system was a 4-channel “Box in a Box” design shown in Figure 37. The LEDs and photodiode detectors are mounted in an aluminum block that is attached to a thermal electric cooler. This insulated box is mounted in another thermal electric cooled insulated box. All power supplies were located in a separate box to reduce the load on the thermal electric coolers. This design ensured that the LEDs and photodiode detectors were maintained at a constant temperature during room temperature changes.

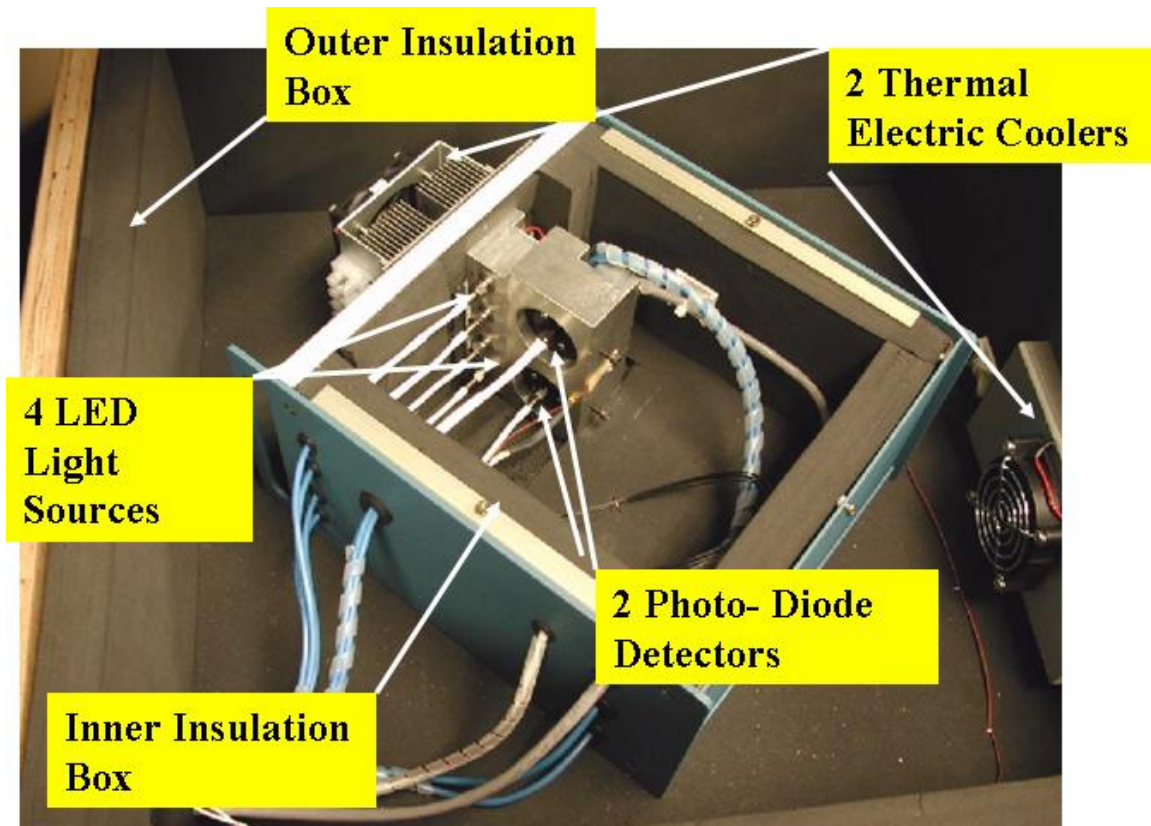


Figure 37. 4-Channel FOHS System

An improved 8-channel FOHS system is shown in Figures 38. Details of the assembly are shown in Figures 39 and 40. The light from the LEDs passes through miniature beam splitters. Part of the light passes through it to a GRIN lens and part of the light is reflected 90° to one of the photodiode detectors. This design greatly increased the light intensity of the system and increased the signal-to-noise ratio.

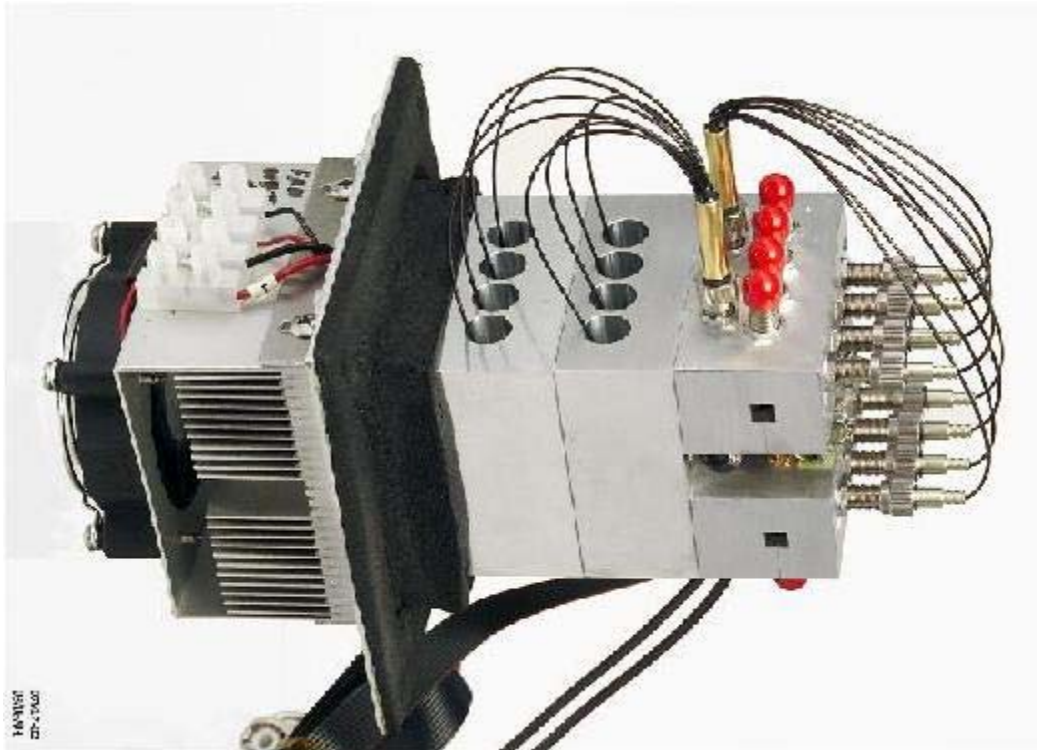


Figure 38. 8-Channel FOHS System Details

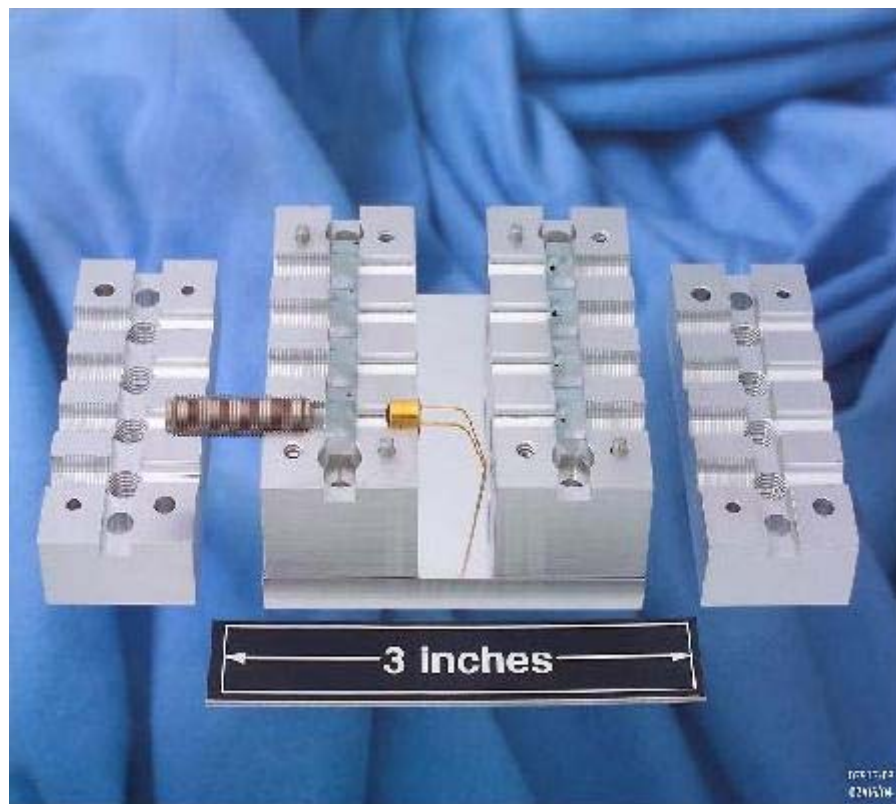


Figure 39. Details of the 8-Channel FOHS System

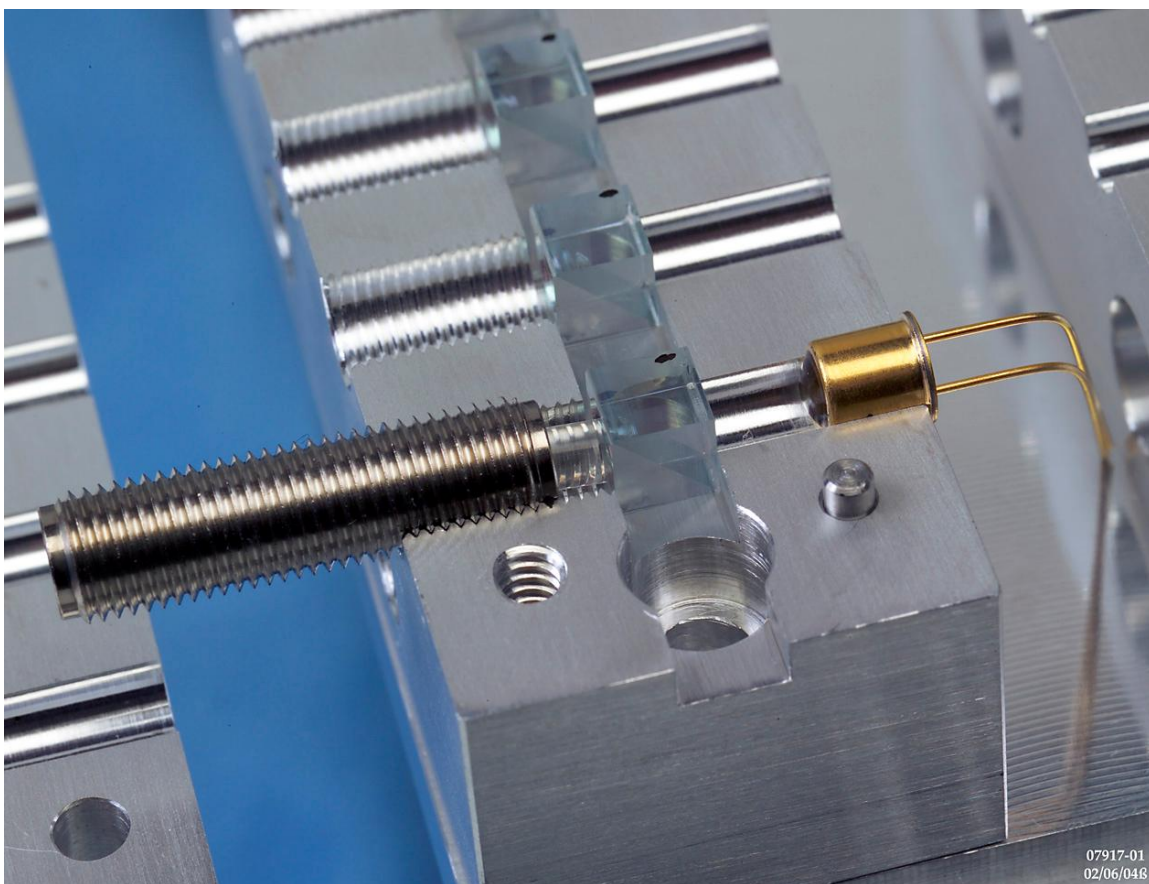


Figure 40. Finer Details of the 8-Channel FOHS System

A complete bench top FOHS System is shown in Figure 41. The LED/photodiode detector assemblies are located to the left in a “Darwin” chamber. This chamber is thermal electrically cooled to maintain constant temperature. The data collection system is located in the front and the oven and vacuum pump are located in the back. The retort used for calibration of up to 8 sensors is shown in Figure 42.



Figure 41. Bench Top FOHS System



Figure 42. Retort for Testing 8 Sensors

In Figure 43 the responses of silicon and germanium detectors are shown. The response of germanium detectors, on the right, shows no effects on temperature changes over the range of the LED light output of 775 to 975 nm. This type of detector was chosen for the FOHS system.

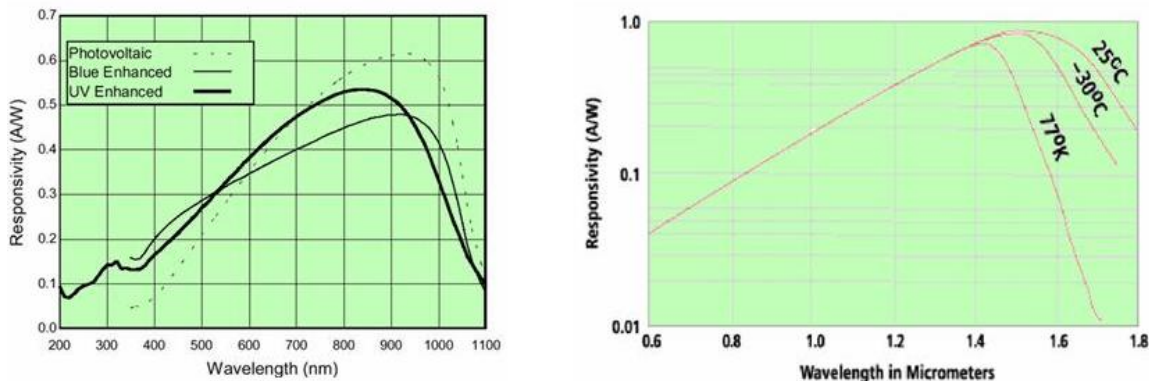


Figure 43. Silicon and Germanium Detector Responses

Calibration

A constant leak hydrogen source was used to perform calibration on sensors. The raw data from the tests, % transmission vs. time, were plotted to ensure that the system responded to hydrogen as expected. Because the baratron data fluctuates, linear behavior was assumed for the hydrogen pressure present throughout the test duration. This linear assumption of pressure increase eliminated the erroneous effects of baratron fluctuation on data analysis. Calibration curves, a plot of the change of % transmission vs. pressure, were fitted to a logarithmic equation. The results showed repeatability throughout the three runs at each temperature. At 40°C, for example, the calibration curves developed from one test were applied to the other two tests in order to predict the system response to the controlled amount of hydrogen over time. The calibration equation accurately predicted the response for all three tests at the same temperature, especially in the lower pressure region. In Figure 44 a calibration run at 70°C for 8 sensors is shown.

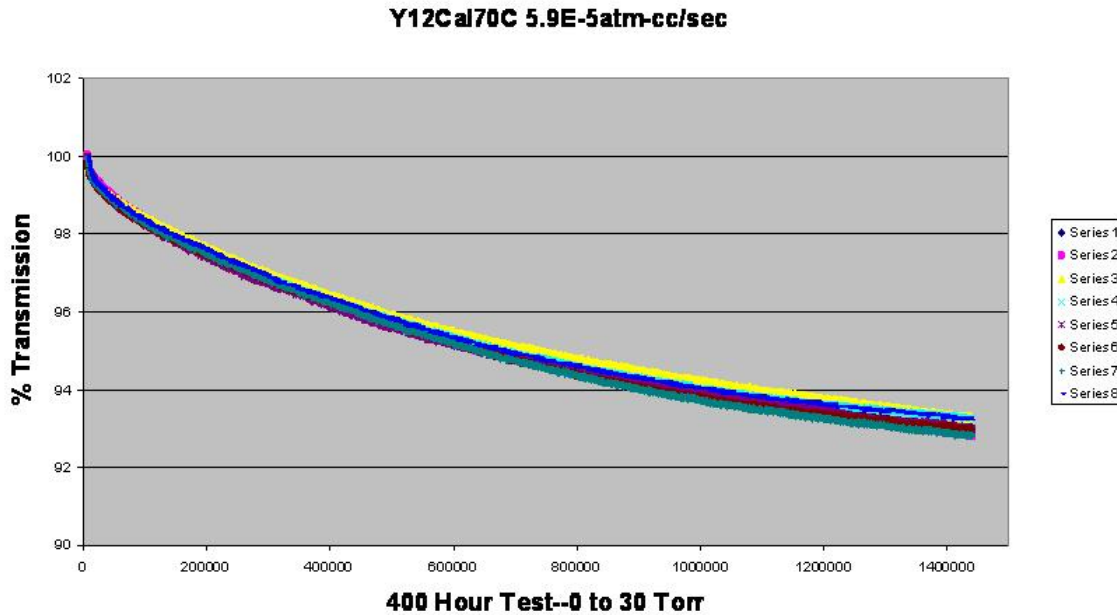


Figure 44. 400-Hour Test

This calibration run demonstrates the stability and repeatability between channels for the final FOHS System. Further details on the calibration will be discussed in the final report on the ESC project, KCP-613-6977.²

Conclusions

Room temperature changes were found to be the cause of most drift and noise problems with the hydrogen sensor system. The use of fiber optic spectrometers was abandoned due these problems and a FOHS system was found to be more stable for this application.

Accomplishments

The FOHS system was redesigned and has eliminated the effects of room temperature changes to its response. The capacity of the system was increased from 4 channels to 8 channels and could be increased further. The use of bulkhead SMA 905 connectors was eliminated except inside of the temperature-controlled FOHS system. The lens holder was redesigned for use with off-the-shelf standard VCR components. This eliminated the need for manually focusing the lens. A modeling simulation was used to verify that the new design was insensitive to thermal changes. The lens coatings were more thoroughly characterized and several tests were established for monitoring production variability. A commercial source of coated lenses was established and response output was more than doubled.

References

1. *Optical Sensor Technology Development and Deployment KC10; Chemically Amplified Optical Sensors LL20* (Topical Report) UNCLASSIFIED. Federal Manufacturing & Technologies: KCP-613-6365, October 2000.
2. *Chemically Amplified Optical Sensor* (Final Report) UNCLASSIFIED. Federal Manufacturing & Technologies: KCP-613-6977, in preparation.